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(54) 【発明の名称】 ポリカーボネート樹脂組成物、並びにこれにて形成された光学記録媒体用基板及び光学記録媒体

(57) 【要約】

【課題】 高密度記録に適した光学記録媒体用基板を与えるポリカーボネート樹脂組成物を提供する。

【解決手段】 アルカリ金属以外の金属の含有量が0.5ppm以下であり、かつ離型剤として多価アルコールの脂肪酸エステルを100~1000ppm含有していることを特徴とするポリカーボネート樹脂組成物。

## 【特許請求の範囲】

【請求項1】 アルカリ金属以外の金属の含有量が0.5ppm以下であり、かつ離型剤として多価アルコールの脂肪酸エステルを100~1000ppm含有していることを特徴とするポリカーボネート樹脂組成物。

【請求項2】 鉄、ニッケル、クロム及びアルミニウムの合計含有量が0.5ppm以下であり、かつ離型剤として多価アルコールの脂肪酸エステルを100~1000ppm含有していることを特徴とするポリカーボネート樹脂組成物。

【請求項3】 離型剤として多価アルコールの脂肪酸エステルを100~1000ppm含有しているポリカーボネート樹脂組成物を射出成形して得られた光学記録媒体用基板であって、アルカリ金属以外の他の金属の含有量が0.5ppm以下であり、かつ成形に供した樹脂組成物中に離型剤として配合されている多価アルコールの脂肪酸エステルの50%以上が残存していることを特徴とする光学記録媒体用基板。

【請求項4】 離型剤として多価アルコールの脂肪酸エステルを100~1000ppm含有しているポリカーボネート樹脂組成物を射出成形して得られた光学記録媒体用基板であって、鉄、ニッケル、クロム及びアルミニウムの合計含有量が0.5ppm以下であり、アルカリ金属以外の他の金属の含有量は0.5ppm以下であり、かつ成形に供した樹脂組成物中に離型剤として配合されている多価アルコール脂肪酸エステルの50%以上が残存していることを特徴とする光学記録媒体用基板。

【請求項5】 ピッチが2.0μm以下のトラックを有していることを特徴とする請求項3又は4記載の光学記録媒体用基板。

【請求項6】 請求項3ないし5のいずれかに記載の光学記録媒体用基板を用いた光学記録媒体。

## 【発明の詳細な説明】

## 【0001】

【発明の属する技術分野】 本発明はポリカーボネート樹脂組成物、特に射出成形により光学記録媒体用基板のような、微細な凹凸を有する成形品を製造するのに好適な、ポリカーボネート樹脂組成物に関するものである。また本発明は、このポリカーボネート樹脂組成物から製造された光学記録媒体用基板、及びこれを用いた光学記録媒体に関するものである。

## 【0002】

【従来の技術】 非接触で情報の読み出しが可能な光学記録方式は、従来の磁気記録方式に比較して、傷や汚れに対して強いという特徴を有している。この方式の記録媒体は、透明基板上に情報記録層を備えた構造を有している。透明基板の素材としては、主にポリカーボネート樹脂が用いられている。ポリカーボネート樹脂は、溶融成形時の耐熱性及び成形後の寸法安定性に優れており、かつ機械的特性も優れているので、光学記録媒体の素材と

して好適であるとされている。光学記録媒体用基板には情報記録用のトラックが設けられているが、記録容量の増大に伴い、このトラックの溝の間隔すなわちトラックのピッチはより狭くなり、また溝の深さはより深くなりつつある。そしてこれに対応して、射出成形により光学記録媒体用基板を成形する際の成形温度や金型温度は、より高温になりつつある。しかし成形温度を高くすると離型性が悪化して、成形品にそりが発生し易くなるという重大な問題がある。そのある光学記録媒体用基板を用いて製造した光学記録媒体は、その部分でビットエラーを生じ易いので、エラー発生率が高くなる。

## 【0003】

【発明が解決しようとする課題】 上記のような事情の下に、成形温度や金型温度が高くても、光記録媒体用基板に要求される特性である、複屈折、複写性及びそりなどの点で優れた製品を与える、離型性のよいポリカーボネート樹脂組成物が求められている。従来から離型剤として多価アルコールの脂肪酸エステルを配合したポリカーボネート樹脂組成物が離型性に優れていることが知られているが、高記録容量の光学記録媒体用基板を射出成形により製造するための高い成形温度及び金型温度の下では、このものは所期の良好な離型性を発現しない。従って本発明は、高記録容量の光学記録媒体用基板の製造に好適な、高い成形温度及び金型温度の下でも良好な離型性を発現する、ポリカーボネート樹脂組成物を提供せんとするものである。また本発明はこのようなポリカーボネート樹脂組成物で成形した光学記録媒体用基板、及びこの基板を用いた光学記録媒体を提供せんとするものである。

## 【0004】

【課題を解決するための手段】 本発明者らの検討によれば多価アルコールの脂肪酸エステルを配合したポリカーボネート樹脂組成物が、高い成形温度及び金型温度の下で所期の離型性を発現しないのは、成形に際し多価アルコールの脂肪酸エステルが熱により他の形態に変化してしまうためである。そしてこの変化は、ポリカーボネートの製造工程やそれに続く加工工程で、装置を構成する金属材料から混入する鉄その他の金属により、著るしく促進されることが判明した。また、多価アルコールの脂肪酸エステルの配合量にも適正值があり、樹脂組成物中における多価アルコールの脂肪酸エステルの含有量が100~1000ppmより多くても少なくとも、射出成形により得られた板に成形欠陥を生じ易いことが判明した。

【0005】 本発明はこのような知見に基づいて達成されたもので、本発明に係るポリカーボネート樹脂組成物は、離型剤として多価アルコールの脂肪酸エステルを100~1000ppm含有し、かつ溶融成形に際して離型剤の熱変化を促進する鉄その他の装置から混入する金属の含有量が著るしく少ないことを特徴とするものであ

る。また、本発明に係る光学記録媒体用基板は、上記のポリカーボネート樹脂組成物の射出成形により製造されたものであって、鉄その他の離型剤の熱変化を促進する金属の含有量が著しく少なく、かつ成形に供した樹脂組成物中の離型剤である多価アルコールの脂肪酸エステル50%以上が残存していることを特徴とするものである。

#### 【0006】

【発明の実施の形態】本発明について詳細に説明すると、本発明に係るポリカーボネート樹脂組成物は、任意のポリカーボネート樹脂に離型剤を配合することにより製造される。ポリカーボネートの原料であるフェノール性化合物としては、ヒドロキノン、レゾルシン、ジヒドロキシジフェニル、ビス(ヒドロキシフェニル)-アルカン、ビス(ヒドロキシフェニル)-シクロアルカン、ビス(ヒドロキシフェニル)-スルフィド、ビス(ヒドロキシフェニル)-エーテル、ビス(ヒドロキシフェニル)-ケトン、ビス(ヒドロキシフェニル)-スルホン、ビス(ヒドロキシフェニル)-スルフォキシド、ビス(ヒドロキシフェニル)-ベンゼン、及び核にアルキル基やハロゲン原子などが置換しているこれらの誘導体などが挙げられる。特に好適なフェノール性化合物としては、2, 2-ビス(4-ヒドロキシフェニル)-プロパン、2, 2-ビス(3, 5-ジメチル-4-ヒドロキシフェニル)-プロパン、1, 1-ビス(4-ヒドロキシフェニル)-シクロヘキサン及び1, 1-ビス(4-ヒドロキシフェニル)-3, 3, 5-トリメチルシクロヘキサンなどが挙げられる。また、3個以上のフェノール性水酸基を有するものをこれらのフェノール性化合物に少量併用して、分岐を有するポリカーボネートを得ることもできる。

【0007】これらのフェノール性化合物からポリカーボネートを製造するのは、ホスゲンを用いる界面重縮合法、又はジフェニルカーボネートを用いる溶融法のいずれの方法によることもできる。得られるポリカーボネートの平均分子量(M<sub>v</sub>)は通常 $1 \times 10^4 \sim 1 \times 10^5$ 程度であるが、光学記録媒体用基板の製造に用いるには、13000~20000、特に14000~18000の範囲であるのが好ましい。

【0008】ポリカーボネートの製造に際しては、使用する原料及び装置から金属が混入しないように細心の注意を払う必要がある。例えばホスゲンを用いる界面重縮合法による場合には、ホスゲンの配管は、腐蝕を防止するため完全に乾燥させてからホスゲンを通すことが必要であり、かつホスゲンは活性炭と接触させて含まれているかもしれない金属成分を除去してから、反応に供するのが好ましい。またアルカリ水溶液の配管は、管壁からの金属成分の溶出を防止するため、耐蝕性に富むものを用いることが必要である。本発明者らの検討によれば、ポリカーボネート中に普通に検出される金属のうちアル

カリ金属は離型剤である多価アルコールの脂肪酸エステルが熱により変質するのを促進する作用は比較的弱い。鉄やクロム、ニッケル、アルミニウムなど、配管や機器の壁面から混入してくる金属は離型剤の熱変化を促進する作用が著しく大きい。そしてポリカーボネート中に通常有意量で見出されるのはこれらの金属なので、通常はこれらの合計を指標として、その含有量が0.5 ppm以下のものを用いる。好ましくは、上記で述べた金属を含めアルカリ金属以外の金属の含有量が0.5 ppm以下のものを用いる。樹脂組成物中の金属含有量は少ないほど好ましく、鉄、クロム、ニッケル及びアルミニウムの合計量が0.1 ppm以下、特に0.05 ppm以下のものを用いると更に好ましい。

【0009】金属の含有量が多いと、射出成形に際し成形温度及び金型温度を高くすると、離型剤として含有させた多価アルコールの脂肪酸エステルが不完全エステルから完全エステルその他にすみやかに熱変化して、得られる成形体中に殆んど残存しなくなる。そして例えばグリセリンモノエステルがグリセリントリエステルに変化すると、離型剤としての作用は著しく低下する。離型剤の多価アルコールの脂肪酸エステルは100~1000 ppmとなるようにポリカーボネートに配合することが必要である。この離型剤の量が過少であると離型性が不十分となり、成形品にそりなどの欠陥が生じ易い。逆にこの量が過多であると、成形時にガスを生じ易く、このガスがスタンパーに付着して転写不良を生じ易い。離型剤の好ましい配合量は100~600 ppm、特に150~500 ppmである。多価アルコールの脂肪酸エステルとしては、グリセリン、トリメチロールプロパン、ヘキサントリオールのような3価のアルコールや、ペンタエリスリトール、メソエリスリトール、キシリトール、ソルビトールのような4価以上の多価アルコールと、炭素数10~30程度の脂肪酸との不完全エステルが用いられる。脂肪酸としてはカプリン酸、ウンデカン酸、ラウリン酸、トリデカン酸、ミリスチン酸、ペンタデカン酸、パルミチン酸、マルガリン酸、ステアリン酸、ノナデカン酸、エイコサン酸、ベヘン酸などが好ましい。これらの多価アルコールと脂肪酸とのエステルとしては、通常はグリセリンモノステアレート、グリセリンモノパルミテート、グリセリンモノミリステート、グリセリンモノラウレートなどのグリセリンモノエステル、ペンタエリスリトールジステアレート、ペンタエリスリトールトリステアレート、ペンタエリスリトールモノパルミテート、ペンタエリスリトールジパルミテート、ペンタエリスリトールトリパルミテート、メソエリスリトールトリラウレート、キシリトールジステアレート、キシリトールトリステアレート、キシリトールテトラステアレートなどが用いられる。これらのエステルはいくつかを併用することもできる。

【0010】本発明に係るポリカーボネート樹脂組成物

は射出成形、押出成形などによって種々の成形品、例えばフィルム、糸、板などに加工することができる。照明器具用部品や光学機器用部品、例えば灯火のハウジングや光学レンズなどにも好適に加工することができる。これらの加工に際しては、所望により常用の種々の添加剤、例えば安定剤、帯電防止剤、燃焼遅延剤などを添加することもできる。本発明に係るポリカーボネート樹脂組成物の特に好適な用途は、射出成形による光学記録媒体用基板の製造である。光学記録媒体としては再生専用型光ディスク（CD、CD-ROM、DVD-ROM等）、記録再生型光ディスク（ライトワンス型；CD-R、DVD-R等）、記録、再生、消去、再書込可能型光ディスク（リライタブル型；MO、CD-RW、DVD-RAM等）があり、いずれもポリカーボネート樹脂組成物の射出成形により基板の製造が行われている。これらのディスクのトラックピッチは記録密度により異なるが、高記録密度のものでは2.0 $\mu$ m以下のトラックピッチが要求されており、将来的には更に狭いピッチ、例えば1.8 $\mu$ m以下のものが要求されるものと考えられるが、このようなディスクの基板を射出成形により製造する場合には特に離型性の良いことが必要なので、本発明に係る樹脂組成物は好適である。

【0011】本発明に係るポリカーボネート樹脂組成物を用いて光学記録媒体用の基板を射出成形する場合に、成形温度は300～400℃、特に320～380℃が好ましい。また、一般に光学記録媒体用基板の射出成形に際しては、押出機の先端に金属繊維から成るフィルターを設置して樹脂中の異物を除去するが、フィルターを構成する金属繊維の表面が化学変化して金属化合物になっていると、この金属化合物がここを通過する溶融樹脂中に混入し、樹脂に含有させてある離型剤の熱変化を促進することがある。従ってフィルターは十分に洗浄して、金属繊維の表面に存在するかもしれない金属化合物

表

\*物を除去してから用いることが望ましい。本発明に係るポリカーボネート樹脂組成物を用いる場合でも、成形過程で金属が混入するなどして組成物中の離型剤である多価アルコールの脂肪酸エステルが50%以上が熱変化するような条件で射出成形したのでは、離型むらや転写不良などの成形不良を生じ易い。

【0012】本発明に係るポリカーボネート樹脂組成物を用いて、上記したところに従い、添加した離型剤である多価アルコールの脂肪酸エステルが50%以上がそのままの形態で残存するような条件で製造した光学記録媒体用基板は、後記する実施例と比較例との対比からも明らかのように、成形欠陥が無くかつ基板特性に優れているので、これに常法により記録層を形成することにより高品質の光学記録媒体とすることができる。

【0013】

【実施例】以下に実施例により本発明を更に具体的に説明するが、本発明はこれらの実施例に限定されるものではない。

ポリカーボネートの製造；苛性ソーダ水溶液、塩化メチレン及びビスフェノールAを混合し、この混合液中にホスゲンを供給してポリカーボネートのオリゴマーを生成させた。なお、触媒としてはトリエチルアミンを用い、分子量調節剤としてはセブチルフェノールを用いた。水相と油相とを分離し、得られた油相に塩化メチレン、苛性ソーダ水溶液及びトリエチルアミンを添加して、窒素雰囲気下、30℃で反応させてポリカーボネートを生成させた。水相と油相とを分離し、油相を希塩酸で洗浄し、次いで排水中に塩素イオンが検出されなくなるまで純水で洗浄した。得られた精製ポリカーボネート溶液をニーダーで粉化して、ポリカーボネートの粉末を得た。得られたポリカーボネートの物性を表-1に示す。

【0014】

【表1】

1

ポリカーボネート	平均分子量 (Mv) *1	分子量分布 (Mw/Mn) *2	Q値 (ml/秒) *3	金属含有量 (ppm) *4
A	15000	2.67	40 $\times 10^{-3}$	0.1
B	21100	2.75	7.1 $\times 10^{-3}$	0.16
C	15600	2.66	34.3 $\times 10^{-3}$	0.09
D	15000	2.67	40 $\times 10^{-3}$	1

【0015】\*1 濃度Cが0.6g/dl塩化メチレン溶液について、20℃で測定した比粘度（ $\eta_{sp}$ ）から、下記式により算出。

【0016】

※【数1】 $\eta_{sp}/C = [\eta] (1 + 0.28\eta_{sp})$   
 $[\eta] = 1.23 \times 10^{-5} \times Mv \times 0.83$

【0017】\*2 ゲルパーミエーションクロマトグラ

※50 フ（東ソー社製品、HLC-8020）を用いて測定。

東ソー社のTSK 5000HLX、4000HLX、3000HLX及び2000HLXをそれぞれ充填した4本のカラムを用い、テトラヒドロフランを溶離液とし、検出は屈折率計で行った。得られたチャートからポリスチレン換算のMw及びMnを求めた。

\*3 120℃で5時間乾燥したポリカーボネート2gを、高化式フローテスター（島津製作所製品 CFT-500、オリフィスは直径1mm、長さ10mm）の280℃に加熱されたオリフィスに仕込んだ。10分後に完全に溶融したのを確認したのち、160kgの荷重下で上記オリフィス中を流下させ、4mm～7mm流下した時間を読み取り、流下容積/時間（ml/秒）で表示\*

シリンダー温度	350℃
金型温度	103℃/98℃
スタンパー	230MB（ISO/IEC 13963規格）
充填時間	0.34秒
冷却時間	4.5秒
圧縮力（トン）	22.5-13.8-10

【0019】得られた基板について下記の項目をチェックした。結果を表-2に示す。

成形欠陥（ピットずれ）；1000枚毎に3枚の基板を取出し、内外周のSFP部及び外周7.8.9バンドを光学顕微鏡で観察した。離型性の不十分な場合、及びスタンパーに揮発物が付着した場合のいずれにもピットずれが発生する。

成形欠陥（離型むら）；1000枚毎に25枚の基板を取出し、反射光及び透過光で目視観察した。離型性が良くない場合には離型むらが発生する。

【0020】複屈折；自動複屈折測定装置（オーク製作所製 ADR-130N）を用いて、面内複屈折及び垂直複屈折を測定した。結果を最大値と最小値とで表示した。

機械特性；光ディスクの規格（ISO/IEC 13963）に従って評価し、十分に余裕をもって合格するものを○、余裕は無いが合格するものを△とした。

脂肪酸エステル含有量；試料を塩化メチレンに溶解し、※

\*した。

\*4 鉄、ニッケル、クロム及びアルミニウムの合計量（これ以外の金属は殆んど含まれていない）。分析は試料を灰化し、ICP分析法により行った。

【0018】実施例1～4及び比較例1～2

ポリカーボネート粉末に多価アルコールの脂肪酸エステルを粉末で配合し、よく混合したのち二軸押出機で窒素雰囲気下、290℃で混練してペレット化した。このペレットを射出成形機（住友重機械社製品 DISK3）に供給し、下記条件で光学記録媒体用の3.5インチ基板を5000枚製造した。

※これにピリジンとトリメチルシリルクロライドを加えて  
20 脂肪酸エステル中の水酸基を全てトリメチルシリル化した。次いで反応液をガスクロマトグラフィーで分析し、添加した脂肪酸エステルの形態で存在しているものを定量した。

【0021】比較例1～6

ポリカーボネート粉末に多価アルコールの脂肪酸エステルと金属化合物とを添加した以外は実施例と同様にしてペレットを製造し、次いでこのペレットを用いて実施例と同様にして光ディスク基板を製造した。結果を表-2に示す。

30 比較例7

ポリカーボネート粉末をそのまま実施例と同様にしてペレット化し、次いでこのペレットを用いて実施例と同様にして光ディスク基板を製造した。結果を表-2に示す。

【0022】

【表2】

表 - 2

		実施例 1	実施例 2	実施例 3	実施例 4
ポリカーボネート		A	B	C	C
脂肪酸エステル	種 類	GMS	GMS	PDS	PDS
	添加量 (ppm)	600	600	600	1000
金属化合物	種 類	-	-	-	-
	添加量 (ppm)	-	-	-	-
ペレット中の脂肪酸エステル(ppm)		580	560	590	900
基板中の添加脂肪酸エステル(ppm)		580	550	580	890
成形欠陥 (ビットずれ)		無	無	無	無
成形欠陥 (離型むら)		無	無	無	無
基板特性 (面内複屈折)		$0 \sim 7 \times 10^{-6}$	-	$-2 \sim 4 \times 10^{-6}$	$0 \sim 7 \times 10^{-6}$
基板特性 (垂直複屈折)		$4.6 \sim 4.9 \times 10^{-4}$	-	$4.7 \sim 5 \times 10^{-4}$	$4.6 \sim 4.9 \times 10^{-4}$
基板特性 (機械特性)		○	-	○	○

【0023】

\* \* 【表3】

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表 - 2 (つづき)

	比較例	比較例	比較例	比較例	比較例	比較例	比較例
	1	2	3	4	5	6	7
ポリカーボネート	D	A	A	A	A	A	A
脂肪酸エステル	GMS	GMS	GMS	GMS	GMS	GMS	-
添加量 (ppm)	600	2000	600	600	600	600	-
金属化合物	-	-	FeCl <sub>3</sub>	TiCl <sub>4</sub>	Ni(OH) <sub>2</sub>	CrCl <sub>3</sub> ·6H <sub>2</sub> O	-
添加量 (ppm)	-	-	3	5	2	5	-
シート中の脂肪酸エステル	80	1850	20	0	10	0	0
基板中の添加脂肪酸エステル (ppm)	75	1800	20	0	10	0	0
成形欠陥 (ピットずれ)	有	有	有	有	有	有	有
成形欠陥 (離型むら)	成形初期に有	無	外周部に有	外周部に有	外周部に有	外周部に有	離型不可
基板特性 (面内複屈折)	-4 ~ -8 × 10 <sup>-6</sup>	-3 ~ -7 × 10 <sup>-6</sup>	-2 ~ -6 × 10 <sup>-6</sup>	-2 ~ -6 × 10 <sup>-6</sup>	0 ~ 8 × 10 <sup>-6</sup>	0 ~ 8 × 10 <sup>-6</sup>	測定不可
基板特性 (垂直複屈折)	4.6 ~ 5.2 × 10 <sup>-4</sup>	4.6 ~ 5.2 × 10 <sup>-4</sup>	4.3 ~ 4.9 × 10 <sup>-4</sup>	4.4 ~ 5.0 × 10 <sup>-4</sup>	4.65 ~ 4.95 × 10 <sup>-4</sup>	4.65 ~ 4.95 × 10 <sup>-4</sup>	測定不可
基板特性 (機械特性)	△	△	△	△	△	△	測定不可

GMS : グリセリンモノステアレート

PDS : ペンタエリスリトールジパルミテート

フロントページの続き

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**(54) POLYCARBONATE RESIN COMPOSITION, SUBSTRATE FOR OPTICAL RECORDING MEDIUM FORMED THEREFROM AND OPTICAL RECORDING MEDIUM**

**(57)Abstract:**

PROBLEM TO BE SOLVED: To obtain a polycarbonate resin composition useful for the production of a substrate for an optical recording medium having a large memory capacity, and manifesting good mold-releasing properties at a high molding temperature and mold temperature by including a metal without an alkali metal in a specific amount or less, and a fatty acid ester of a polyhydric alcohol.

SOLUTION: The objective polycarbonate resin composition contains  $\leq 0.5$  ppm metal without an alkali metal, and 100-1,000 ppm fatty acid ester of a polyhydric alcohol as a mold releasing agent. The composition is obtained by compounding the mold releasing agent with a polycarbonate resin. 2,2-Bis(4-hydroxyphenyl)propane or the like is cited as a phenolic compound of a raw material of the polycarbonate. Glycerol monostearate or the like is usable as the fatty acid ester of the polyhydric alcohol.

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CLAIMS

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[Claim(s)]

[Claim 1] The polycarbonate resin constituent which the content of metals other than alkali metal is 0.5 ppm or less, and is characterized by containing 100-1000 ppm of fatty acid ester of polyhydric alcohol as a release agent.

[Claim 2] The polycarbonate resin constituent which the sum total content of iron, nickel, chromium, and aluminum is 0.5 ppm or less, and is characterized by containing 100-1000 ppm of fatty acid ester of polyhydric alcohol as a release agent.

[Claim 3] The substrate for optical record media characterized by 50% or more of the fatty acid ester of the polyhydric alcohol blended as a release agent into the resin constituent with which it is the substrate for optical record media which injection molded the polycarbonate resin constituent which contains 100-1000 ppm of fatty acid ester of polyhydric alcohol as a release agent, and was obtained, and the content of other metals other than alkali metal is 0.5 ppm or less, and shaping was presented remaining.

[Claim 4] It is the substrate for optical record media which injection molded the polycarbonate resin constituent which contains 100-1000 ppm of fatty acid ester of polyhydric alcohol as a release agent, and was obtained. The sum total content of iron, nickel, chromium, and aluminum is 0.5 ppm or less. The content of other metals other than alkali metal is a substrate for optical record media characterized by 50% or more of the polyhydric-alcohol fatty acid ester blended as a release agent into the resin constituent with which is 0.5 ppm or less and shaping was presented remaining.

[Claim 5] The substrate for optical record media according to claim 3 or 4 characterized by the pitch having the truck 2.0 micrometers or less.

[Claim 6] The optical record medium using the substrate for optical record media according to claim 3 to 5.

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[Translation done.]

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DETAILED DESCRIPTION

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## [Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to a suitable polycarbonate resin constituent to manufacture a polycarbonate resin constituent, especially the mold goods which have detailed irregularity like the substrate for optical record media with injection molding. Moreover, this invention relates to the substrate for optical record media manufactured from this polycarbonate resin constituent, and the optical record medium using this.

[0002]

[Description of the Prior Art] The optical recording method which information can read has the description of being strong, to a blemish or dirt as compared with the conventional magnetic-recording method by non-contact. The record medium of this method has the structure equipped with the information recording layer on the transparence substrate. As a raw material of a transparence substrate, polycarbonate resin is mainly used. Since polycarbonate resin is excellent in the thermal resistance at the time of melting shaping, and the dimensional stability after shaping and the mechanical property is also excellent, it is made suitable as a raw material of an optical record medium. Although the truck for information record is established in the substrate for optical record media, with buildup of storage capacity, spacing of the slot of this truck, i.e., the pitch of a truck, becomes narrower, and a tooth depth is becoming deeper. And corresponding to this, whenever [ molding-temperature metallurgy mold temperature / at the time of fabricating the substrate for optical record media with injection molding ] is becoming an elevated temperature more. However, if molding temperature is made high, a mold-release characteristic will get worse, and the serious problem of becoming easy to generate camber is in mold goods. Since the optical record medium manufactured using the substrate for optical record media with camber tends to produce a bit error in the part of camber, an error incidence rate becomes high.

[0003]

[Problem(s) to be Solved by the Invention] The bottom of the above situations is asked for the good polycarbonate resin constituent of a mold-release characteristic which gives the product which was excellent in respect of the birefringence and copy nature which are a property required of the substrate for optical recording media, camber, etc. even if whenever molding-temperature metallurgy mold temperature ] is high. Although it is known that the polycarbonate resin constituent which blended the fatty acid ester of polyhydric alcohol as a release agent from the former is excellent in a mold-release characteristic, under a high molding temperature and the high die temperature for manufacturing the substrate for optical record media of high storage capacity with injection molding, this thing does not discover an expected good mold-release characteristic. Therefore, this invention uses as an offer plug the polycarbonate resin constituent which discovers a good mold-release characteristic also under a suitable high molding temperature and the suitable die temperature for manufacture of the substrate for optical record media of high storage capacity. Moreover, this invention uses as an offer plug the substrate for optical record media fabricated with such a polycarbonate resin constituent, and the optical record medium using this substrate.

[0004]

[Means for Solving the Problem] The polycarbonate resin constituent which blended the fatty acid ester of polyhydric alcohol according to examination of this invention persons does not discover a mold-release characteristic expected in the bottom of a high molding temperature and a die temperature for the fatty acid ester of polyhydric alcohol changing with heat to other gestalten on the occasion of shaping. And this change is a processing process following the production process of a polycarbonate, or it, and it became clear that \*\*\*\*\* acceleration was carried out with the metal of the iron mixed from the metallic material which constitutes equipment, and others. Moreover, there was a proper value also in the loadings of the fatty acid ester of polyhydric alcohol, and even if there were more contents of the fatty acid ester of the polyhydric alcohol in a resin constituent than 100-1000 ppm, it became clear that it was easy to produce a shaping defect to the plate obtained by injection molding at least.

[0005] the content of the metal mixed from the equipment of the iron which this invention was attained based on such knowledge, and the polycarbonate resin constituent concerning this invention contains 100-1000 ppm of fatty acid ester of polyhydric alcohol as a release agent, and promotes the thermal change of a release agent on the occasion of melting shaping, and others -- \*\*\*\*\* -- it is characterized by few things. Moreover, the substrate for optical record media concerning this invention has remarkably few contents of the metal which is manufactured by injection molding of the above-mentioned polycarbonate resin constituent, and promotes the thermal change of the release agent of iron and others, and is characterized by 50% or more of the fatty acid ester of the polyhydric alcohol which is a release agent in the resin constituent with which shaping was presented remaining.

[0006]

[Embodiment of the Invention] If this invention is explained to a detail, the polycarbonate resin constituent concerning this invention will be manufactured by blending a release agent with the polycarbonate resin of arbitration. As a phenol nature compound which is the raw material of a polycarbonate A hydroquinone, resorcinol, dihydroxydiphenyl, a screw (hydroxyphenyl)-alkane, Screw (hydroxyphenyl)-cycloalkane, a screw (hydroxyphenyl)-sulfide, The screw (hydroxyphenyl)-ether, a screw (hydroxyphenyl)-ketone, Screw (hydroxyphenyl)-sulfone, screw (hydroxyphenyl)-sulfoxide, screw (hydroxyphenyl)-benzene, these derivatives that the alkyl group, the halogen atom, etc. have permuted by the nucleus are mentioned. As an especially suitable phenol nature compound, it is 2 and 2-screw (4-hydroxyphenyl)-propane, 2, and 2-screw (3, 5-dimethyl-4-hydroxyphenyl)-propane, 1, and 1-screw (4-hydroxyphenyl)-cyclohexane and 1, and 1-screw (4-hydroxyphenyl). - A 3, 3, and 5-trimethyl cyclohexane etc. is mentioned. Moreover, little concomitant use of what has three or more phenolic hydroxyl groups can be carried out at these phenol nature compounds, and the polycarbonate which has branching can also be obtained.

[0007] Manufacturing a polycarbonate from these phenol nature compounds can also be based on which approach of the interfacial polycondensation method for using a phosgene, or the scorification using diphenyl carbonate. The average molecular weight (Mv) of the polycarbonate obtained is usually  $1 \times 10^4$  to  $1 \times 10^5$ . Although it is extent, in order to use for manufacture of the substrate for optical record media, it is desirable 13000-20000, and that it is especially the range of 14000-18000.

[0008] It is necessary to pay careful attention so that a metal may not mix on the occasion of manufacture of a polycarbonate from the raw material to be used and equipment. For example, when based on the interfacial polycondensation method using a phosgene, piping of a phosgene needs to let a phosgene pass, after making it dry thoroughly, in order to prevent corrosion, and it is desirable [ a phosgene ] to present a reaction, after a phosgene removes the metal component which may be contacted to activated carbon and may be contained. Moreover, piping of an alkali water solution needs to use what is rich in corrosion resistance in order to prevent elution of the metal component from a tube wall. the operation whose metal mixed from the wall surface of piping, such as iron, chromium, nickel, and aluminum, or a device the operation which promotes that, as for alkali metal, the fatty acid ester of the polyhydric alcohol which is a release agent deteriorates with heat among the metals ordinarily detected in a polycarbonate is comparatively weak according to examination of this invention persons, but promotes the thermal change of a release agent -- \*\*\*\*\* -- it is large. And since these metals are usually found out by the significant quantity in a polycarbonate, the content usually uses a thing 0.5 ppm or less by making these sum totals into an index. Preferably, the contents of metals other than alkali metal including the metal described above use a thing 0.5 ppm or less. Few metal contents in a resin constituent are so desirable that there are, and 0.1 ppm or less, when the total quantity of iron, chromium, nickel, and aluminum uses especially a thing 0.05 ppm or less, it is still more desirable.

[0009] If there are many metaled contents and molding temperature and a die temperature will be made high on the occasion of injection molding, the fatty acid ester of the polyhydric alcohol made to contain as a release agent will carry out a thermal change for whether being Sumiya at perfect ester and others from imperfection ester, and will not carry out \*\*\*\*\* survival into the Plastic solid acquired. And if glycerol monoester changes to glycerol triester, for example, the operation as a release agent will fall remarkably. The fatty acid ester of the polyhydric alcohol of a release agent needs to blend with a polycarbonate so that it may be set to 100-1000 ppm. A mold-release characteristic becomes [ too little / the amount of this release agent / inadequate ], and it is easy to produce defects, such as camber, in mold goods. Conversely, it is easy to produce gas as this amount being excessive at the time of shaping, and this gas adheres to a stamper and tends to produce a poor imprint. 100-600 ppm especially of desirable loadings of a release agent are 150-500 ppm. As fatty acid ester of polyhydric alcohol, the imperfection ester of trivalent alcohol like a glycerol, trimethylol propane, and hexane triol, the polyhydric alcohol more than tetravalence like pentaerythritol, meso erythritol, xylitol, and a sorbitol, and a with a carbon number of about ten to 30 fatty acid is used. As a fatty acid, a capric acid, undecanoic acid, a lauric acid, a tridecane acid, a myristic acid, a pentadecane acid, a palmitic acid, margaric acid, stearin acid, a nonadecane acid, eicosanoic acid, behenic acid, etc. are desirable. As ester of these polyhydric alcohol and fatty acids Usually, glycerol monostearate, glycerol monopalmitate, Glycerol monoester, such as a glycerol mono-millimeter state and glycerine monolaurate, Pentaerythritol distearate, pentaerythritol tristearate, Pentaerythritol monopalmitate, pentaerythritol dipalmitate, Pentaerythritol tripalmitate, meso erythritol trilaurate, xylitol distearate, xylitol tristearate, xylitol tetra-stearate, etc. are used. These ester can also use some together.

[0010] The polycarbonate resin constituent concerning this invention is processible into various mold goods, for example, a film, yarn, a plate, etc. with injection molding, extrusion molding, etc. It is processible suitable for the components for luminaires and the components for optical instruments, for example, housing, an optical lens, etc. of a lamp. On the occasion of these processings, various additives in ordinary use, for example, a stabilizer, an antistatic agent, a combustion retarder, etc. can also be added by request. Especially the suitable application of the polycarbonate resin constituent concerning this invention is manufacture of the substrate for optical record media by injection molding. As an optical record medium, there are mold optical disks (rewritable type; MO, CD-RW, DVD-RAM, etc.) which can be rewritten [ the mold optical disks only for playbacks (CD, CD-ROM, DVD-ROM, etc.), record playback mold optical disks (write-once mold; CD-R DVD-R, etc.), record, playback, elimination, and ], and manufacture of a substrate is performed for all by injection molding of a polycarbonate resin constituent. Although it is thought that the thing of high recording density requires the track pitch 2.0 micrometers or less, and a prospective still narrower pitch, for example, a thing 1.8 micrometers or less, is required although the track pitch of these disks changes with recording density, since the good thing of a mold-release characteristic is the need especially when manufacturing the substrate of such a disk with injection molding, the resin constituent concerning this invention is suitable.

[0011] When injection molding the substrate for optical record media using the polycarbonate resin constituent concerning this

invention, especially molding temperature has desirable 320-380 degrees C 300-400 degrees C. Moreover, although the filter which consists at the head of an extruder of a metal fiber is installed and the foreign matter in resin is generally removed on the occasion of injection molding of the substrate for optical record media, when the front face of the metal fiber which constitutes a filter changes chemically and it has become metallic compounds, these metallic compounds may mix into the melting resin which passes through this, and may promote the thermal change of the release agent which resin is made to have contained. Therefore, as for a filter, it is desirable to fully wash, and to use, after removing the metallic compounds which may exist on the surface of a metal fiber. Even when using the polycarbonate resin constituent concerning this invention, in having injection molded on conditions in which a metal mixes by the forming process and 50% or more of the fatty acid ester of the polyhydric alcohol which is a release agent in a constituent carries out a thermal change, it is easy to produce poor shaping, such as mold release unevenness and a poor imprint.

[0012] The above-mentioned place is followed using the polycarbonate resin constituent concerning this invention. The substrate for optical record media manufactured on conditions which remain with a gestalt with 50% or more as it is [ the fatty acid ester of the polyhydric alcohol which is the added release agent ] Since there is no shaping defect and it excels in the substrate property so that clearly also from comparison with the example and the example of a comparison which carry out a postscript, it can consider as the optical record medium of high quality by forming a recording layer in this with a conventional method.

[0013]

[Example] Although an example explains this invention still more concretely below, this invention is not limited to these examples.

Manufacture of a polycarbonate; a caustic-alkali-of-sodium water solution, a methylene chloride, and bisphenol A were mixed, the phosgene was supplied into this mixed liquor, and the oligomer of a polycarbonate was made to generate. In addition, t-butylphenol was used as a molecular-weight modifier, using triethylamine as a catalyst. The aqueous phase and an oil phase are separated, a methylene chloride, a caustic-alkali-of-sodium water solution, and triethylamine are added to the obtained oil phase, it was made to react at 30 degrees C, and the polycarbonate was made to generate under nitrogen-gas-atmosphere mind. The aqueous phase and an oil phase were separated, dilute hydrochloric acid washed the oil phase, and pure water washed until the chloride ion was no longer detected subsequently to under wastewater. The obtained purification polycarbonate solution was powdered by the kneader, and the powder of a polycarbonate was obtained. The physical properties of the obtained polycarbonate are shown in a table -1.

[0014]

[A table 1]

表 1

ポリカーボネート	平均分子量 (Mv) *1	分子量分布 (Mw/Mn) **	Q値 (ml/秒) **	金属含有量 (ppm)**
A	15000	2.67	$40 \times 10^{-3}$	0.1
B	21100	2.75	$7.1 \times 10^{-2}$	0.16
C	15600	2.66	$34.8 \times 10^{-3}$	0.09
D	15000	2.67	$40 \times 10^{-3}$	1

[0015] \*1 Concentration C computes by the following formula about a 0.6 g/dl methylene chloride solution from the specific viscosity (etaSP) measured at 20 degrees C.

[0016]

[Equation 1]  $\text{etaSP}/C = [\text{eta}] (1 + 0.28\text{etaSP})$

$[\text{eta}] = 1.23 \times 10^{-5} \text{Mv} \times 0.83$  [0017] \*2 Measure using a gel permeation chromatograph (the TOSOH CORP. product, HLC-8020). TSK of TOSOH CORP. Using four columns filled up with 5000HLX, 4000HLX, 3000HLX, and 2000HLX(s), respectively, the tetrahydrofuran was made into the eluate and the refractive-index meter performed detection. Mw and Mn of polystyrene conversion were calculated from the obtained chart.

\*3 Polycarbonate 2g dried at 120 degree C for 5 hours was taught to the orifice heated by 280 degrees C of a high-sized type flow tester (500 Shimadzu product CFT- an orifice the diameter of 1mm, die length of 10mm). After checking having fused thoroughly after 10 minutes, the time amount which was made to flow down in the above-mentioned orifice, and flowed down 4mm - 7mm under the 160kg load was read, and it displayed by the flowing-down volume / time amount (ml/second).

\*4 The total quantity of iron, nickel, chromium, and aluminum (there are no metals other than this \*\*\*\*\* rare \*\*\*\*). Analysis ashed the sample and was performed with the ICP analysis method.

[0018] The fatty acid ester of polyhydric alcohol was blended with examples 1-4 and the example 1 of a comparison - 2 polycarbonate powder with powder, and after often mixing, it kneaded and pelletized at 290 degrees C under nitrogen-gas-atmosphere mind with the twin screw extruder. This pellet was supplied to the injection molding machine

(Sumitomo heavy machinery company product DISK3), and 5000 3.5 inch substrates for optical record media were manufactured on the following conditions.

Cylinder temperature 350 degrees C Die temperature 103 degrees C / 98 degrees C Stamper 230MB (ISO/IEC 13963 specification)

Injection time 0.34 seconds Cooldown delay 4.5 seconds Compressive force (ton) 22.5-13.8-10 [0019] The following item was checked about the obtained substrate. A result is shown in a table -2.

Shaping defect (pit gap); the SFP section of drawing and an inside-and-outside periphery and periphery 7.8.9 band were observed for three substrates with the optical microscope every 1000 sheets. When a mold-release characteristic is inadequate, a pit gap occurs in all when volatile matter adheres to a stamper.

Shaping defect (mold release unevenness); visual observation of the 25 substrates was carried out by drawing, the reflected light, and the transmitted light every 1000 sheets. When a mold-release characteristic is not good, mold release unevenness occurs.

[0020] Birefringence; the birefringence within a field and the vertical birefringence were measured using the automatic birefringence measuring device (ORC Manufacturing make ADR-130N). The result was expressed as maximum and the minimum value.

Mechanical characteristic; it evaluated according to the specification (ISO/IEC 13963) of an optical disk, and what passes although there are not O and allowances about what fully passes with allowances was made into \*\*.

Fatty-acid-ester content; the sample was dissolved in the methylene chloride, a pyridine and trimethylsilyl chloride were added to this, and all the hydroxyl groups in fatty acid ester were trimethylsilylated. Subsequently, the quantum of what analyzes reaction mixture with a gas chromatography and exists with the gestalt of the added fatty acid ester was carried out.

[0021] Except having added the fatty acid ester and metallic compounds of polyhydric alcohol to the example 1 of a comparison - 6 polycarbonate powder, the pellet was manufactured like the example and, subsequently the optical disk substrate was manufactured like the example using this pellet. A result is shown in a table -2.

Example of comparison 7 polycarbonate powder was pelletized like the example as it was, and, subsequently the optical disk substrate was manufactured like the example using this pellet. A result is shown in a table -2.

[0022]

[A table 2]

表 - 2

		実施例 1	実施例 2	実施例 3	実施例 4
ポリカーボネート		A	B	C	C
脂肪酸エステル	種 類	GMS	GMS	PDS	PDS
	添加量 (ppm)	800	800	600	1000
金属化合物	種 類	—	—	—	—
	添加量 (ppm)	—	—	—	—
ペレット中の脂肪酸エステル(ppm)		580	560	590	900
基板中の添加脂肪酸エステル(ppm)		580	550	580	890
成形欠陥 (ピットずれ)		無	無	無	無
成形欠陥 (離型むら)		無	無	無	無
基板特性 (面内複屈折)		$0 \sim 7 \times 10^{-6}$	—	$-2 \sim 4 \times 10^{-6}$	$0 \sim 7 \times 10^{-6}$
基板特性 (垂直複屈折)		$4.6 \sim 4.9 \times 10^{-4}$	—	$4.7 \sim 5 \times 10^{-4}$	$4.6 \sim 4.9 \times 10^{-4}$
基板特性 (機械特性)		○	—	○	○

[0023]

[A table 3]

表 1-2 (つぎ)

	比較例	比較例	比較例	比較例	比較例	比較例	比較例
	1	2	3	4	5	6	7
ポリカーボネート	D	A	A	A	A	A	A
脂肪族エステル 種類 添加量 (ppm)	GMS 600	GMS 2000	GMS 600	GMS 600	GMS 600	GMS 600	- -
金属化合物 種類 添加量 (ppm)	- -	- -	FeCl <sub>3</sub> 3	TiCl <sub>4</sub> 5	Ni(OH) <sub>2</sub> 2	CrCl <sub>3</sub> ·6H <sub>2</sub> O 5	- -
ペレット中の難防蝕成分 (%)	80	1850	20	0	10	0	0
基板中の添加難防蝕成分 (%)	75	1800	20	0	10	0	0
成形欠陥 (ピットずれ)	有	有	有	有	有	有	有
成形欠陥 (霧むら)	成形初期に有 $-4 \sim -8 \times 10^{-6}$	無 $-3 \sim -7 \times 10^{-6}$	外周部に有 $-2 \sim -6 \times 10^{-6}$	外周部に有 $-2 \sim -6 \times 10^{-6}$	外周部に有 $0 \sim -8 \times 10^{-6}$	外周部に有 $0 \sim -8 \times 10^{-6}$	離型不可
基板特性 (面内複屈折)	$4.6 \sim 5.2 \times 10^{-4}$	$4.6 \sim 5.2 \times 10^{-4}$	$4.3 \sim 4.9 \times 10^{-4}$	$4.4 \sim 5.0 \times 10^{-4}$	$4.65 \sim 4.95 \times 10^{-4}$	$4.65 \sim 4.95 \times 10^{-4}$	測定不可
基板特性 (垂直複屈折)							測定不可
基板特性 (機械特性)	△	△	△	△	△	△	測定不可

GMS: グリセリンモノステアレート

PDS:ペンタエリスリトールジハルミテート

[Translation done.]

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to a suitable polycarbonate resin constituent to manufacture a polycarbonate resin constituent, especially the mold goods which have detailed irregularity like the substrate for optical record media with injection molding. Moreover, this invention relates to the substrate for optical record media manufactured from this polycarbonate resin constituent, and the optical record medium using this.

[0002]

[Description of the Prior Art] The optical recording method which information can read has the description of being strong, to a blemish or dirt as compared with the conventional magnetic-recording method by non-contact. The record medium of this method has the structure equipped with the information recording layer on the transparence substrate. As a material of a transparence substrate, polycarbonate resin is mainly used. Since polycarbonate resin is excellent in the thermal resistance at the time of melting shaping, and the dimensional stability after shaping and the mechanical property is also excellent, it is made suitable as a material of an optical record medium. Although the truck for information record is established in the substrate for optical record media, with increase of storage capacity, spacing of the slot of this truck, i.e., the pitch of a truck, becomes narrower, and the depth of flute is becoming deeper. And corresponding to this, whenever [ molding-temperature metallurgy mold temperature / at the time of fabricating the substrate for optical record media with injection molding ] is becoming an elevated temperature more. However, if molding temperature is made high, a mold-release characteristic will get worse, and the serious problem of becoming easy to generate camber is in mold goods. Since the optical record medium manufactured using the substrate for optical record media with camber tends to produce a bit error in the part of camber, an error incidence rate becomes high.

[0003]

[Problem(s) to be Solved by the Invention] The bottom of the above situations is asked for the good polycarbonate resin constituent of a mold-release characteristic which gives the product which was excellent in respect of the birefringence and copy nature which are a property required of the substrate for optical recording media, camber, etc. even if whenever [ molding-temperature metallurgy mold temperature ] is high. Although it is known that the polycarbonate resin constituent which blended the fatty acid ester of polyhydric alcohol as a release agent from the former is excellent in a mold-release characteristic, under a high molding temperature and the high die temperature for manufacturing the substrate for optical record media of high storage capacity with injection molding, this thing does not discover an expected good mold-release characteristic. Therefore, this invention uses as an offer plug the polycarbonate resin constituent which discovers a good mold-release characteristic also under a suitable high molding temperature and the suitable die temperature for manufacture of the substrate for

optical record media of high storage capacity. Moreover, this invention uses as an offer plug the substrate for optical record media fabricated with such a polycarbonate resin constituent, and the optical record medium using this substrate.

[0004]

[Means for Solving the Problem] The polycarbonate resin constituent which blended the fatty acid ester of polyhydric alcohol according to examination of this invention persons does not discover a mold-release characteristic expected in the bottom of a high molding temperature and a die temperature for the fatty acid ester of polyhydric alcohol changing with heat to other gestalten on the occasion of shaping. And this change is a processing process following the production process of a polycarbonate, or it, and it became clear that \*\*\*\*\* promotion was carried out with the metal of the iron mixed from the metallic material which constitutes equipment, and others. Moreover, there was a proper value also in the loadings of the fatty acid ester of polyhydric alcohol, and even if there were more contents of the fatty acid ester of the polyhydric alcohol in a resin constituent than 100-1000 ppm, it became clear that it was easy to produce a shaping defect to the plate obtained by injection molding at least.

[0005] the content of the metal mixed from the equipment of the iron which this invention was attained based on such knowledge, and the polycarbonate resin constituent concerning this invention contains 100-1000 ppm of fatty acid ester of polyhydric alcohol as a release agent, and promotes the thermal change of a release agent on the occasion of melting shaping, and others -- \*\*\*\*\* -- it is characterized by few things. Moreover, the substrate for optical record media concerning this invention has remarkably few contents of the metal which is manufactured by injection molding of the above-mentioned polycarbonate resin constituent, and promotes the thermal change of the release agent of iron and others, and is characterized by 50% or more of the fatty acid ester of the polyhydric alcohol which is a release agent in the resin constituent with which shaping was presented remaining.

[0006]

[Embodiment of the Invention] If this invention is explained to a detail, the polycarbonate resin constituent concerning this invention will be manufactured by blending a release agent with the polycarbonate resin of arbitration. As a phenol nature compound which is the raw material of a polycarbonate A hydroquinone, resorcinol, dihydroxydiphenyl, a screw (hydroxyphenyl)-alkane, Screw (hydroxyphenyl)-cycloalkane, a screw (hydroxyphenyl)-sulfide, The screw (hydroxyphenyl)-ether, a screw (hydroxyphenyl)-ketone, Screw (hydroxyphenyl)-sulfone, screw (hydroxyphenyl)-sulfoxide, screw (hydroxyphenyl)-benzene, these derivatives that the alkyl group, the halogen atom, etc. have permuted by the nucleus are mentioned. As an especially suitable phenol nature compound, it is 2 and 2-screw (4-hydroxyphenyl)-propane, 2, and 2-screw (3, 5-dimethyl-4-hydroxyphenyl)-propane, 1, and 1-screw (4-hydroxyphenyl)-cyclohexane and 1, and 1-screw (4-hydroxyphenyl). - A 3, 3, and 5-trimethyl cyclohexane etc. is mentioned. Moreover, little concomitant use of what has three or more phenolic hydroxyl groups can be carried out at these phenol nature compounds, and the polycarbonate which has branching can also be obtained.

[0007] Manufacturing a polycarbonate from these phenol nature compounds can also be based on which approach of the interfacial polycondensation method for using a phosgene, or the scorification using diphenyl carbonate. The average molecular weight (Mv) of the polycarbonate obtained is usually  $1 \times 10^4$  to  $1 \times 10^5$ . Although it is extent, in order to use for manufacture of the substrate for optical record media, it is desirable 13000-20000, and that it is especially the range of 14000-18000.

[0008] It is necessary to pay careful attention so that a metal may not mix on the occasion of manufacture of a polycarbonate from the raw material to be used and equipment. For example, when based on the interfacial polycondensation method using a phosgene, piping of a phosgene needs to let a phosgene pass, after making it dry completely, in order to prevent



corrosion, and it is desirable [ a phosgene ] to present a reaction, after a phosgene removes the metal component which may be contacted to activated carbon and may be contained. Moreover, piping of an alkali water solution needs to use what is rich in corrosion resistance in order to prevent the elution of the metal component from a tube wall. the operation whose metal mixed from the wall surface of piping, such as iron, chromium, nickel, and aluminum, or a device the operation which promotes that, as for alkali metal, the fatty acid ester of the polyhydric alcohol which is a release agent deteriorates with heat among the metals ordinarily detected in a polycarbonate is comparatively weak according to examination of this invention persons, but promotes the thermal change of a release agent -- \*\*\*\*\* -- it is large. And since these metals are usually found out by the significant quantity in a polycarbonate, the content usually uses a thing 0.5 ppm or less by making these sum totals into an index. Preferably, the contents of metals other than alkali metal including the metal described above use a thing 0.5 ppm or less. Few metal contents in a resin constituent are so desirable that there are, and 0.1 ppm or less, when the total quantity of iron, chromium, nickel, and aluminum uses especially a thing 0.05 ppm or less, it is still more desirable.

[0009] If there are many metaled contents and molding temperature and a die temperature will be made high on the occasion of injection molding, the fatty acid ester of the polyhydric alcohol made to contain as a release agent will carry out a thermal change for whether being Sumiya at perfect ester and others from imperfect ester, and will not carry out \*\*\*\*\* survival into the Plastic solid acquired. And if glycerol monoester changes to glycerol triester, for example, the operation as a release agent will fall remarkably. The fatty acid ester of the polyhydric alcohol of a release agent needs to blend with a polycarbonate so that it may be set to 100-1000 ppm. A mold-release characteristic becomes [ too little / the amount of this release agent / inadequate ], and it is easy to produce defects, such as camber, in mold goods. Conversely, it is easy to produce gas as this amount being excessive at the time of shaping, and this gas adheres to a stamper and tends to produce a poor imprint. 100-600 ppm especially of desirable loadings of a release agent are 150-500 ppm. As fatty acid ester of polyhydric alcohol, the imperfect ester of trivalent alcohol like a glycerol, trimethylol propane, and hexane triol, the polyhydric alcohol more than tetravalence like pentaerythritol, meso erythritol, xylitol, and a sorbitol, and a with a carbon number of about ten to 30 fatty acid is used. As a fatty acid, a capric acid, undecanoic acid, a lauric acid, a tridecane acid, a myristic acid, a pentadecane acid, a palmitic acid, margaric acid, stearin acid, a nonadecane acid, eicosanoic acid, behenic acid, etc. are desirable. As ester of these polyhydric alcohol and fatty acids Usually, glycerol monostearate, glycerol monopalmitate, Glycerol monoester, such as a glycerol mono-millimeter state and glycerine monolaurate, Pentaerythritol distearate, pentaerythritol tristearate, Pentaerythritol monopalmitate, pentaerythritol dipalmitate, Pentaerythrytol tripalmitate, meso erythritol trilaurate, xylitol distearate, xylitol tristearate, xylitol tetra-stearate, etc. are used. These ester can also use some together.

[0010] The polycarbonate resin constituent concerning this invention is processible into various mold goods, for example, a film, yarn, a plate, etc. with injection molding, extrusion molding, etc. It is processible suitable for the components for luminaires and the components for optical instruments, for example, housing, an optical lens, etc. of lamplight. On the occasion of these processings, various additives in ordinary use, for example, a stabilizer, an antistatic agent, a combustion retarder, etc. can also be added by request. Especially the suitable application of the polycarbonate resin constituent concerning this invention is manufacture of the substrate for optical record media by injection molding. As an optical record medium, there are mold optical disks (rewritable type; MO, CD-RW, DVD-RAM, etc.) which can be rewritten [ the mold optical disks only for playbacks (CD, CD-ROM, DVD-ROM, etc.), record playback mold optical disks (write-once mold; CD-R DVD-R, etc.), record, playback, elimination, and ], and manufacture of a substrate is performed for all by injection molding of a polycarbonate resin

constituent. Although it is thought that the thing of high recording density requires the track pitch 2.0 micrometers or less, and a prospective still narrower pitch, for example, a thing 1.8 micrometers or less, is required although the track pitch of these disks changes with recording density, since the good thing of a mold-release characteristic is the need especially when manufacturing the substrate of such a disk with injection molding, the resin constituent concerning this invention is suitable.

[0011] When carrying out injection molding of the substrate for optical record media using the polycarbonate resin constituent concerning this invention, especially molding temperature has desirable 320-380 degrees C 300-400 degrees C. Moreover, although the filter which consists at the tip of an extruder of a metal fiber is installed and the foreign matter in resin is generally removed on the occasion of injection molding of the substrate for optical record media, when the front face of the metal fiber which constitutes a filter changes chemically and it has become metallic compounds, these metallic compounds may mix into the melting resin which passes through this, and may promote the thermal change of the release agent which resin is made to have contained. Therefore, as for a filter, it is desirable to fully wash, and to use, after removing the metallic compounds which may exist on the surface of a metal fiber. Even when using the polycarbonate resin constituent concerning this invention, in having carried out injection molding on conditions in which a metal mixes by the forming process and 50% or more of the fatty acid ester of the polyhydric alcohol which is a release agent in a constituent carries out a thermal change, it is easy to produce poor shaping, such as mold release unevenness and a poor imprint.

[0012] The above-mentioned place is followed using the polycarbonate resin constituent concerning this invention. The substrate for optical record media manufactured on conditions which remain with a gestalt with 50% or more as it is [ the fatty acid ester of the polyhydric alcohol which is the added release agent ] Since there is no shaping defect and it excels in the substrate property so that clearly also from contrast with the example and the example of a comparison which carry out a postscript, it can consider as the optical record medium of high quality by forming a recording layer in this with a conventional method.

[0013]

[Example] Although an example explains this invention still more concretely below, this invention is not limited to these examples.

Manufacture of a polycarbonate; a caustic-alkali-of-sodium water solution, a methylene chloride, and bisphenol A were mixed, the phosgene was supplied into this mixed liquor, and the oligomer of a polycarbonate was made to generate. In addition, t-butylphenol was used as a molecular-weight modifier, using triethylamine as a catalyst. The aqueous phase and an oil phase are separated, a methylene chloride, a caustic-alkali-of-sodium water solution, and triethylamine are added to the obtained oil phase, it was made to react at 30 degrees C, and the polycarbonate was made to generate under nitrogen-gas-atmosphere mind. The aqueous phase and an oil phase were separated, dilute hydrochloric acid washed the oil phase, and pure water washed until the chlorine ion was no longer detected subsequently to under wastewater. The obtained purification polycarbonate solution was powdered by the kneader, and the powder of a polycarbonate was obtained. The physical properties of the obtained polycarbonate are shown in Table -1.

[0014]

[Table 1]

表 1

ポリカーボネート	平均分子量 (Mv) * <sup>1</sup>	分子量分布 (Mw/Mn) * <sup>2</sup>	Q値 (ml/秒) * <sup>3</sup>	金属含有量 (ppm)* <sup>4</sup>
A	15000	2.67	$40 \times 10^{-2}$	0.1
B	21100	2.75	$7.1 \times 10^{-2}$	0.16
C	15600	2.66	$34.3 \times 10^{-2}$	0.09
D	15000	2.67	$40 \times 10^{-2}$	1

[0015] \*1 Concentration. C computes by the following formula about a 0.6 g/dl methylene chloride solution from the specific viscosity (etaSP) measured at 20 degrees C.

[0016]

[Equation 1]  $\text{etaSP}/C = [\text{eta}] (1 + 0.28\text{etaSP})$

[eta] =  $1.23 \times 10^{-5} M_v \times 0.83$  [0017] \*2 Measure using a gel permeation chromatograph (the TOSOH CORP. product, HLC-8020). TSK of TOSOH CORP. Using four columns filled up with 5000HLX, 4000HLX, 3000HLX, and 2000HLX(s), respectively, the tetrahydrofuran was made into the eluate and the refractive-index meter performed detection. Mw and Mn of polystyrene conversion were calculated from the obtained chart.

\*3 Polycarbonate 2g dried at 120 degree C for 5 hours was taught to the orifice heated by 280 degrees C of a quantity-sized type flow tester (500 Shimadzu product CFT- an orifice the diameter of 1mm, die length of 10mm). After checking having fused completely after 10 minutes, the time amount which was made to flow down in the above-mentioned orifice, and flowed down 4mm - 7mm under the 160kg load was read, and it displayed by the flowing-down volume / time amount (ml/second).

\*4 The total quantity of iron, nickel, chromium, and aluminum (there are no metals other than this \*\*\*\*\* rare \*\*\*\*). Analysis ashed the sample and was performed with the ICP analysis method.

[0018] The fatty acid ester of polyhydric alcohol was blended with examples 1-4 and the example 1 of a comparison - 2 polycarbonate powder with powder, and after often mixing, it kneaded and pelletized at 290 degrees C under nitrogen-gas-atmosphere mind with the twin screw extruder. This pellet was supplied to the injection molding machine (Sumitomo heavy machinery company product DISK3), and 5000 3.5 inch substrates for optical record media were manufactured on the following conditions.

Cylinder temperature 350 degrees C Die temperature 103 degrees C / 98 degrees C Stamper 230MB (ISO/IEC 13963 specification)

Injection time 0.34 seconds Cooldown delay 4.5 seconds Compressive force (ton) 22.5-13.8-10 [0019] The following item was checked about the obtained substrate. A result is shown in Table -2.

Shaping defect (pit gap); the SFP section of drawing and an inside-and-outside periphery and periphery 7.8.9 band were observed for three substrates with the optical microscope every 1000 sheets. When a mold-release characteristic is inadequate, a pit gap occurs in all when volatile matter adheres to a stamper.

Shaping defect (mold release unevenness); visual observation of the 25 substrates was carried out by drawing, the reflected light, and the transmitted light every 1000 sheets. When a

mold-release characteristic is not good, mold release unevenness occurs.

[0020] Birefringence; the birefringence within a field and the perpendicular birefringence were measured using the automatic birefringence measuring device (ORC Manufacturing make ADR-130N). The result was expressed as maximum and the minimum value.

Mechanical characteristic; it evaluated according to the specification (ISO/IEC 13963) of an optical disk, and what passes although there are not O and allowances about what fully passes with allowances was made into \*\*.

Fatty-acid-ester content; the sample was dissolved in the methylene chloride, a pyridine and trimethylsilyl chloride were added to this, and all the hydroxyl groups in fatty acid ester were trimethylsilylated. Subsequently, the quantum of what analyzes reaction mixture with a gas chromatography and exists with the gestalt of the added fatty acid ester was carried out.

[0021] Except having added the fatty acid ester and metallic compounds of polyhydric alcohol to the example 1 of a comparison - 6 polycarbonate powder, the pellet was manufactured like the example and, subsequently the optical disk substrate was manufactured like the example using this pellet. A result is shown in Table -2.

Example of comparison 7 polycarbonate powder was pelletized like the example as it was, and, subsequently the optical disk substrate was manufactured like the example using this pellet. A result is shown in Table -2.

[0022]

[Table 2]

表 - 2

		実施例 1	実施例 2	実施例 3	実施例 4
ポリカーボネート		A	B	C	C
脂肪酸エステル	種 類	GMS	GMS	PDS	PDS
	添加量 (ppm)	600	600	600	1000
金属化合物	種 類	—	—	—	—
	添加量 (ppm)	—	—	—	—
ペレット中の脂肪酸エステル(ppm)		580	560	590	900
基板中の添加脂肪酸エステル(ppm)		580	550	580	890
成形欠陥 (ピットずれ)		無	無	無	無
成形欠陥 (離型むら)		無	無	無	無
基板特性 (面内複屈折)		$0 \sim 7 \times 10^{-6}$	—	$-2 \sim 4 \times 10^{-6}$	$0 \sim 7 \times 10^{-6}$
基板特性 (垂直複屈折)		$4.6 \sim 4.9 \times 10^{-4}$	—	$4.7 \sim 5 \times 10^{-4}$	$4.6 \sim 4.9 \times 10^{-4}$
基板特性 (機械特性)		○	—	○	○

[0023]

[Table 3]

表 - 2 (つづき)

		比較例	比較例	比較例	比較例	比較例	比較例
ポリカーボネート		1	2	3	4	5	6
脂肪酸エステル	種 類	D	A	A	A	A	A
	添加量 (ppm)	GMS 600	GMS 2000	GMS 600	GMS 600	GMS 600	GMS 600
金属化合物	種 類	—	—	FeCl <sub>3</sub>	TiCl <sub>4</sub>	Ni(OH) <sub>2</sub>	CrCl <sub>3</sub> ・6H <sub>2</sub> O
	添加量 (ppm)	—	—	3	5	2	5
ペレット中の脂肪酸エステル(ppm)		80	1850	20	0	10	0
基板中の添加脂肪酸エステル(ppm)		75	1800	20	0	10	0
成形欠陥 (ピットずれ)		有	有	有	有	有	有
成形欠陥 (離型むら)		成形初期に有	無	外周部に有	外周部に有	外周部に有	外周部に有
基板特性 (面内複屈折)		$-4 \sim -8 \times 10^{-6}$	$-3 \sim 7 \times 10^{-6}$	$-2 \sim 6 \times 10^{-6}$	$-2 \sim 6 \times 10^{-6}$	$0 \sim 8 \times 10^{-6}$	$0 \sim 8 \times 10^{-6}$
基板特性 (垂直複屈折)		$4.6 \sim 5.2 \times 10^{-4}$	$4.6 \sim 5.2 \times 10^{-4}$	$4.3 \sim 4.9 \times 10^{-4}$	$4.4 \sim 5.0 \times 10^{-4}$	$4.65 \sim 4.95 \times 10^{-4}$	$4.65 \sim 4.95 \times 10^{-4}$
基板特性 (機械特性)		△	△	△	△	△	△

GMS : グリセリンモノステアレート

PDS : ペンタエリスリトールジパルミテート

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CLAIMS

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[Claim(s)]

[Claim 1] The polycarbonate resin constituent which the content of metals other than alkali metal is 0.5 ppm or less, and is characterized by containing 100-1000 ppm of fatty acid ester of polyhydric alcohol as a release agent.

[Claim 2] The polycarbonate resin constituent which the sum total content of iron, nickel, chromium, and aluminum is 0.5 ppm or less, and is characterized by containing 100-1000 ppm of fatty acid ester of polyhydric alcohol as a release agent.

[Claim 3] The substrate for optical record media characterized by 50% or more of the fatty acid ester of the polyhydric alcohol blended as a release agent into the resin constituent with which it is the substrate for optical record media obtained by carrying out injection molding of the polycarbonate resin constituent which contains 100-1000 ppm of fatty acid ester of polyhydric alcohol as a release agent, and the content of other metals other than alkali metal is 0.5 ppm or less, and shaping was presented remaining.

[Claim 4] It is the substrate for optical record media obtained by carrying out injection molding of the polycarbonate resin constituent which contains 100-1000 ppm of fatty acid ester of polyhydric alcohol as a release agent. The sum total content of iron, nickel, chromium, and aluminum is 0.5 ppm or less. The content of other metals other than alkali metal is a substrate for optical record media characterized by 50% or more of the polyhydric-alcohol fatty acid ester blended as a release agent into the resin constituent with which is 0.5 ppm or less and shaping was presented remaining.

[Claim 5] The substrate for optical record media according to claim 3 or 4 characterized by the pitch having the truck 2.0 micrometers or less.

[Claim 6] The optical record medium using the substrate for optical record media according to claim 3 to 5.

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[Translation done.]